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Investigation of Microstructure of Oxygen-Containing Copper

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Abstract

The results of the study of fine structure of continuous casted bars of oxygen-containing copper produced by casting in the belt water-cooled crystallizer are presented. The character of fractures of samples of copper cast bars in different directions depending on technological parameters of continuous casting was investigated. For determination of chemical composition of samples of cast bars and detection of presence of possible impurities in copper the microspectral analysis was carried out. It is shown that removing of sources of gasing of copper melt leads to decreasing of volume fraction of eutectic Cu-Cu_2O, discontinuity flaws and pores in the structure of cast bars.

Keywords: Copper, Continuos casting, Rolling, Rolled wire, Contirod method, Microstructure, Fracture, Microspectral analysis.

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1. Intorduction

At present continuous methods of manufacture of metallurgical products are prevalent. This methods make it possible considerably improving of productivity and reducing of number of technological operations [1, 2]. The rolled wire of oxygen-containing copper is produced by combined method of casting and rolling Contirod method. The cathode copper of brand M00k as charge material is used [3]. Cathodes are melted in the gas shaft furnace. The melt of copper is transported into the foundry ladle by system of spouts. The continuous casting of copper is carried out by belt water-cooled crystallizer. The cast copper bar of rectangular cross section with dimensions of 120x70 mm is fed in 14-mill rolling mill for production of copper rolled wire with diameter of 8 mm. The detailed manufacturing scheme of Contirod method is shown in the article [4].

The standard method of quality control of copper rolled wire is twist testing with subsequent untwisting of sample of rolled wire of 300 mm long with a 10x10 cycle [5]. After testing the rolled wire is defective in the event that on the surface of rolled wire the cracks with depth more than 0.2 mm are detected. The quality of copper rolled wire depends largely on the quality of continuous cast bar. Such defects as subsurface

blowholes, gas porosity and cold shuts can occur in the cast bar [6, 7]. It is shown in the articles [8, 9] that the macrostructure of continuous cast bar produced by casting in the belt water-cooled crystallizer is characterized by presence of four structural zones which join with five surfaces. For investigation of quality of continuous cast bars the industrial experiment was carried out. During the experiment samples of cast bars were cutted.

2. Materials and Methods

Experiments were carried out with using of industrial line of combined continuous casting and rolling Contirod. The following parameters have been hold during the casting of copper bar of rectangular cross section with 120x70 mm dimensions: casting temperature was from 1120 to 1130 °C, casting speed was from 10,4 to 10,5 m/min, temperature of cooling water for steel belts of crystallizer was from 20 to 26 °C, temperature of bronze blocks of crystallizer was from 100 to 110 °C. Because of continuity of Contirod method the sampling of cast bars was carried out during the scheduled stoppage of rolling mill for preventive inspection. Templates of cast bars were cutted to obtain samples for analysis of microstructure and investigation of character of fractures. The schemes of cutting of templates are shown at Fig. 1.

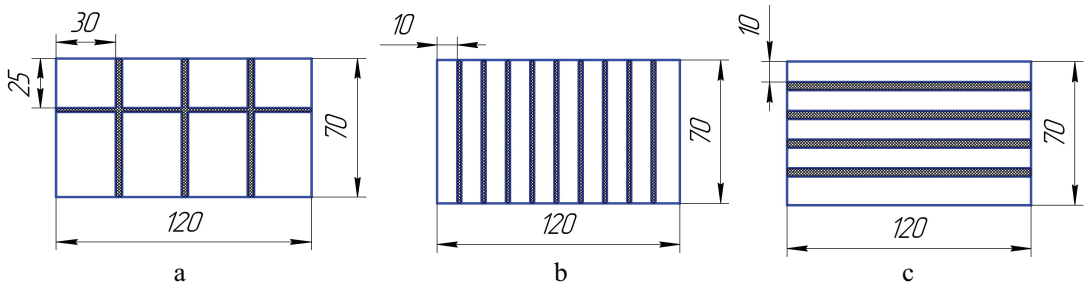


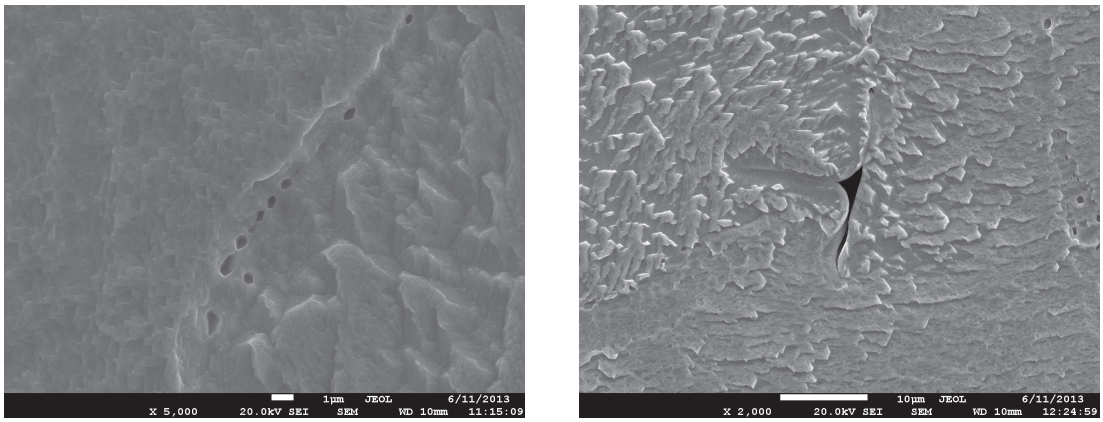
Figure 1: Schemes of cutting of templates of cast bars for analysis of microstructure (a) and investigation of character of fracture in the vertical (b) and horizontal (c) direction.

Samples for analysis of microstructure were grinded using abrasive paper and were polished using diamond paste with following chemical etch using nitric acid. The analysis of microstructure of samples of continuous cast bar of copper with help of scanning electron microscope JEOL JSM-7001F equipped with electron-probe microanalysis device Oxford INCA X-max 80 was carried out. As result energy-dispersion spectrums of samples of copper and maps of distribution of chemical elements were obtained. Moreover the character of fractures of cast bar was studied. To this purpose templates of cast bars were cutted in vertical and horizontal direction. For the purpose of forming of high-quality fracture the incision on the surface of sample was applied. Thereafter the sample was dipped in liquid nitrogen with temperature -196 °C and was crushed with

help of shock testing machine. Samples cutted in vertical direction were crushed along axises of dendritics of first degree. Samples cutted in horizontal direction were crushed perpendicularly to axises of dendritics.

3. Results and Discussion

As example the microstructure of samples of cast copper is shown at Fig 2. It was detected that in the structure of cast copper aggregates of eutectic Cu-Cu_2O (Fig. 2, a) and gas porosity and discontinuity flaws in the junction of crystals (Fig. 2, b) are presented.



a b

Figure 2: The microstructure of continuous cast copper bar: a - aggregates of eutectic Cu-Cu_2O; b - discontinuity flaw in the junction of crystals.

Maps of distribution of chemical elements in samples of cast copper with help of electron-probe microanalysis device were obtained by method of elementwise mapping (Fig. 3).

It is established that in samples of cast bars a copper contains mainly. The segregation of impurities except oxygen in samples of cast copper does not detected. This is edvence that the conent of impurities is lower of detection limit of microanalyzer.

The microstructure of fractures of samples of cast copper is shown at Fig. 4.

The results of analysis of microstructure of copper samples shown that in any case the ductile fracture is observed. In fasets of fracture particles of eutectic Cu-Cu_2O of different sizes are observed clearly. In samples crushed crosswise to axes of dendritics fasets are elongate and secondary cracks on dendritics boundaries are observed. Moreover in samples gas pores are presented.

The main sources of hydrogen saturation of copper during the process of production of rolled wire by Contirod method are following: the injection of air to the connecting

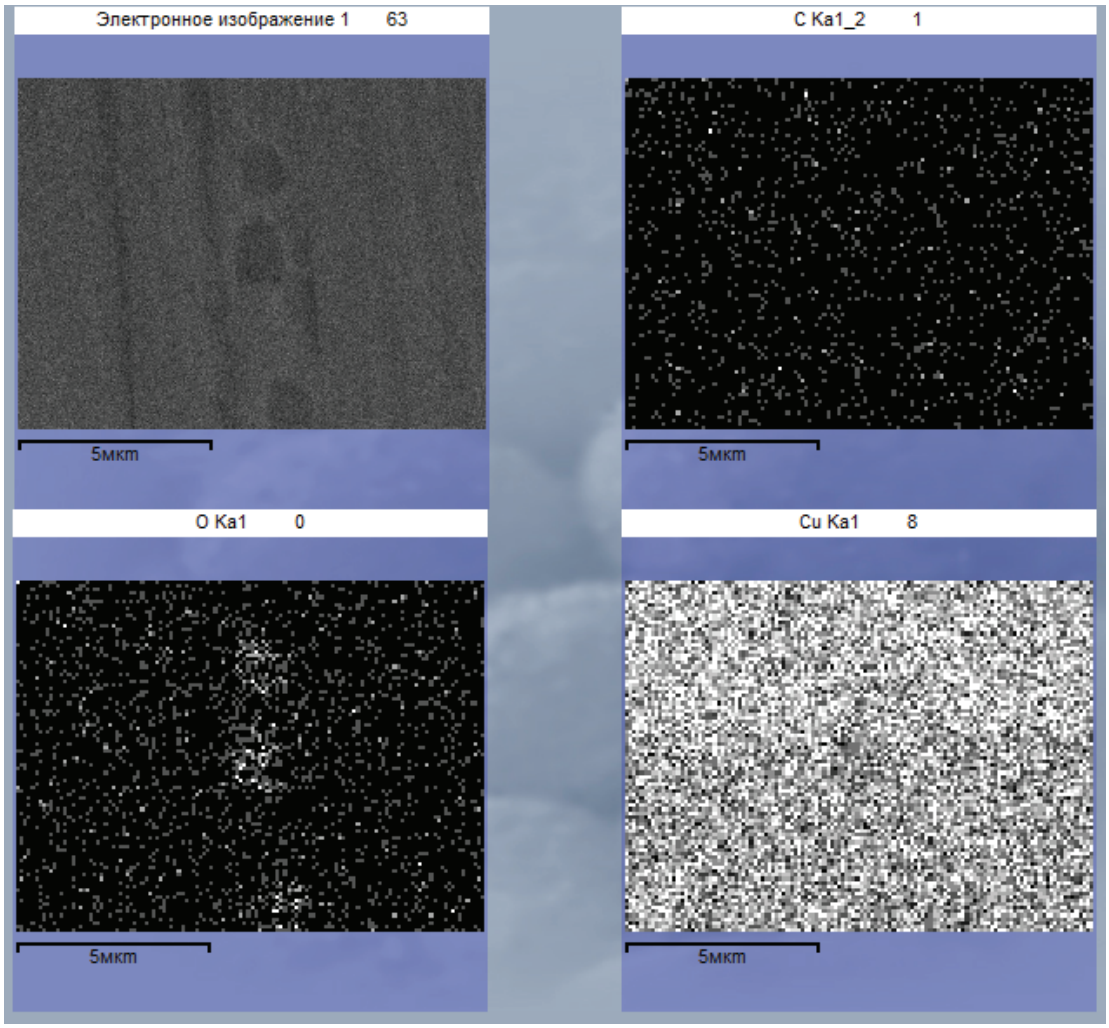
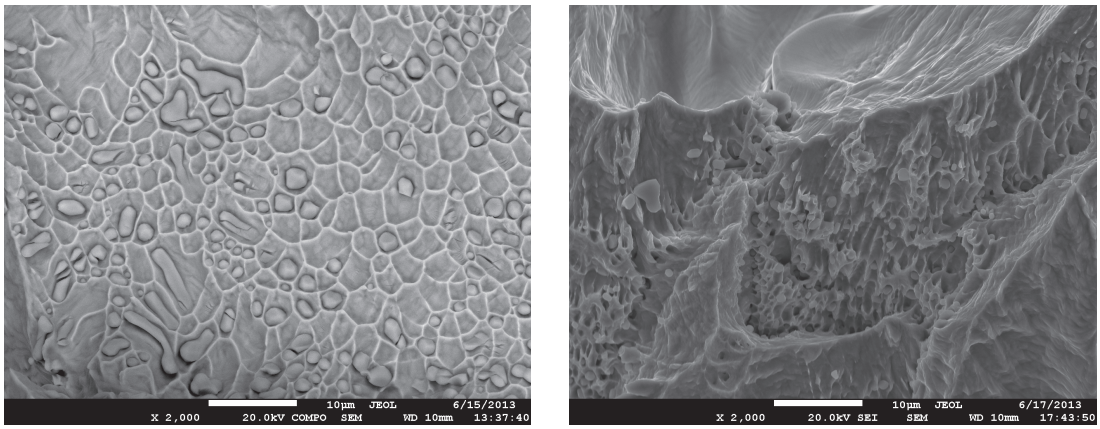


Figure 3: Maps of distribution of chemical elements in samples of cast copper.

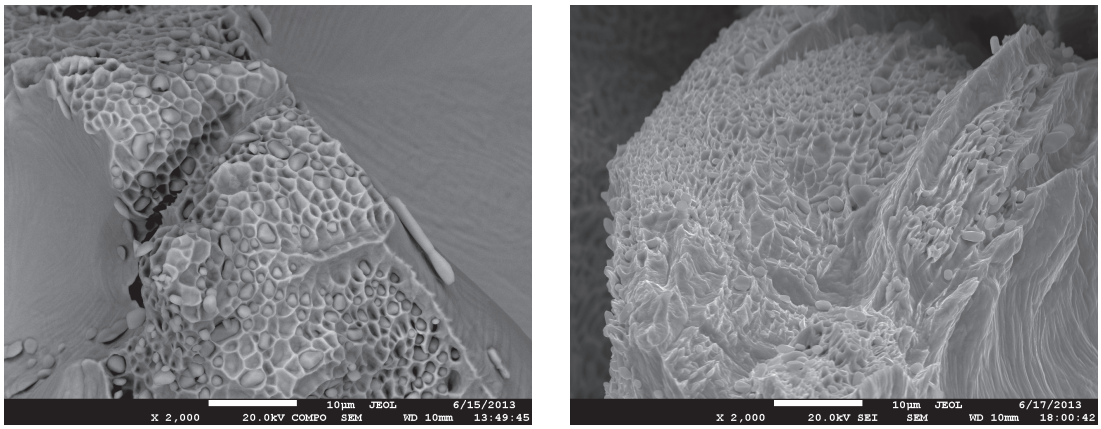


a b

Figure 4: The microstructure of fractures of samples of cast copper: a – in horizontal direction; b – in vertical direction.

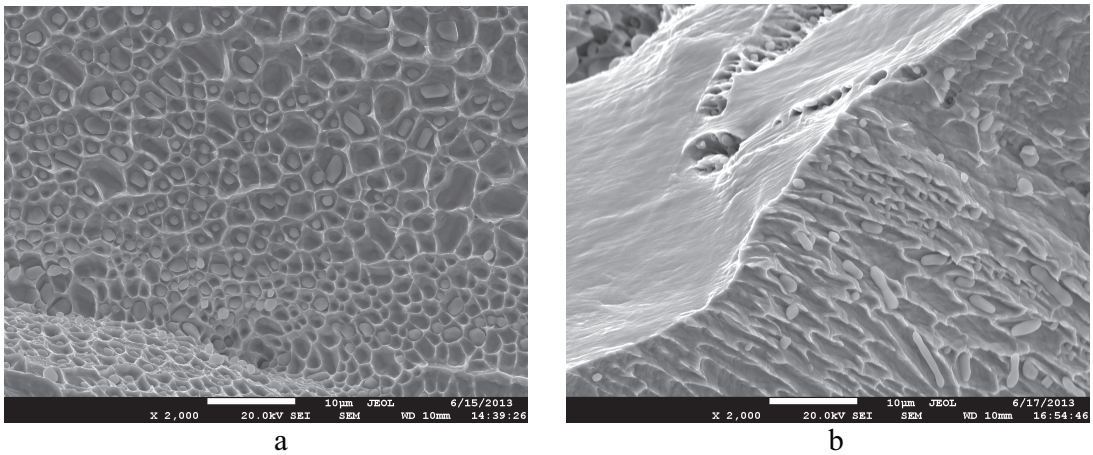
spout in oreder to correct oxygen content in copper and the injection of nitrogen to the

cast spout for the melt level monitoring. For study of influence of parameters of preparation of copper melt to casting on microstructure of fractures of samples of continuous cast bar the series of experiments was carried out. The technological parameters were following: the stopping an air injection to the connecting spout; the stopping a nitrogen injection to the cast spout; the nitrogen of special purity injection for the melt level monitoring. Photos of fractures of horizontal and vertical samples of cast bars produced with using of different technological parameters of preparation of copper melt are shown at Fig. 5-7.



a b

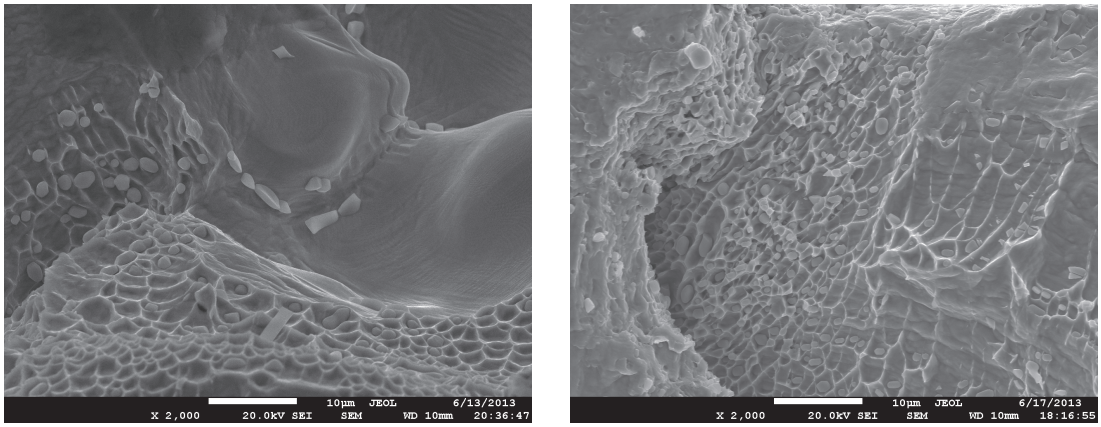
Figure 5: The microstructure of horizontal (a) and vertical (b) samples of cast bar produced without air injection to the connecting spout but with nitrogen injection to the cast spout.



a b

Figure 6: The microstructure of horizontal (a) and vertical (b) samples of cast bar produced without air injection to the connecting spout and nitrogen injection to the cast spout.

The results of analysis of fracture character of samples of cast bars shown that in the samples of cast bars produced by operating technology the quantitu of particles eutectic is very much. The stopping an air injection to the connecting spout leads to reduction in the quantity of particles of eutectic Cu-Cu_2O in the microstructure of fracture. In the fractures of samples cutted from cast bars produced with nitrogen injection to the



a b

Figure 7: The microstructure of horizontal (a) and vertical (b) samples of cast bar produced without air injection to the connecting spout and with nitrogen of special purity injection to the cast spout.

cast spout the large quantity of pores and discontinuity flaws is detected. This defects are stress concentrators and lead to destruction of samples. It is established that the stopping a nitrogen injection to the cast spout as well as the nitrogen of special purity injection for the melt level monitoring leads to reduction of the quantity of defects in the cross section of cast bar. The results of experiments are in accord with earlier studies on investigation of nitrogen content along the casting tract of Contirod line [4].

4. Summary

The results of analysis of microstructure of samples of continuous cast copper shown that in the samples the gas porosity and particles of eutectic Cu-Cu_2O along grains boundaries are presented. The results of microanalysis confirmed the prsence only oxygen in copper. The content of other impurities in copper is lower of detection limit of microanalyzer. The character of fractures of continuous cast copper bars samples in any case is the ductile with the presence of eutectic Cu-Cu_2O in fasets. The eutectic Cu-Cu_2O along grains boundaries and the dissipated gas porosity in continuous cast bar are stress concentrators and can lead to cracks on the surface of rolled wire during twist testing with subsequent untwisting of sample of rolled wire. The results of metallographyc analysis of character of fractures of copper samples shown that the removal of sources of hydrogen saturation of copper during the process of production of rolled wire by Contirod method leads to reduction of volume fraction of eutectic Cu-Cu_2O, pores and discontnuity flaws in continuous cast bars. This fact leads to improvement of quality of copper rolled wire.

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